

CHARACTERIZATION OF AROMATIC FRACTIONS FROM  
NON-PETROLEUM DERIVED JP-5 TYPE FUELS

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Introduction

The Department of Defense has initiated programs aimed at evaluating liquid hydrocarbon fuels which are derived from alternate (fossil) sources: coal, oil shale and tar sands. Of major importance to the Navy are aircraft fuels (JP-5) and ship diesel fuels (DFM). The properties of these kerosene fuels are controlled by means of stringent specifications tests. However, complete knowledge of fuel chemical composition defines the properties of the fuel. Certain properties which are of critical importance to the Navy can be altered by simple physical methods (i.e., flash point (1)). However, other fuel properties depend strongly on fuel composition and therefore, are not subject to facile alteration.

In order to gain a clearer understanding of the effects of chemical composition on fuel properties, a detailed analysis was undertaken. The discussion in this paper will be limited to jet fuels used in Naval aircraft (JP-5). Jet fuels derived from coal, oil shale and tar sands were analyzed. The normal alkane analysis has been reported elsewhere (1). Reported below are the results of analysis of the aromatic components of these fuels.

Production of Fuels

Coal Derived Fuels

The coal derived fuels used in this work were obtained by liquefaction of a Western Kentucky and a Utah coal via the COED process (2). In this process, coal is pyrolyzed in an ebullated bed retort at temperatures ranging from 600 to 1600°F. The condensed oil is hydrotreated over Ni-Mo catalyst (~2500 psig H<sub>2</sub>, 7-800°F) to produce a low sulfur crude oil. The crude oil was then refined by hydrotreatment (3) and distilled to give the JP-5 grade fuels, Sun 1 (Western Kentucky coal), Sun 2 (Utah coal) and Sun 5 (Western Kentucky coal). A schematic of the major process steps used in the production of these fuels is shown in Figure 1.

Alternative methods of refining coal derived crude oils were examined. One such method involved selective removal of heavy aromatics and polar organics (containing N, S, O) by solvent extraction. In a method developed by the Navy (4), a straight run middle distillate from Utah coal derived crude oil (bp 350-550°F) was consecutively treated with sulfuric acid and furfural. The resultant fuel (designated "Sol. Extr.") was used directly without further processing.

Oil Shale Derived Fuel

Green River oil shale was mined and surface retorted by the Paraho Development Corporation. A sample of JP-5 grade kerosene derived from this oil shale was supplied to the Navy by Applied Systems Corporation, under a contract let by the Office of Naval Research. In the Paraho process, crushed and sized oil shale is fed by gravity into a vertical retort which is maintained at 1000°F. Shale oil which was driven from the rock was coked and then fractionated. The middle distillate fraction was hydrotreated and desulfurized in two stages. The major process steps employed are shown schematically in Figure 2. The refining of this batch of shale oil has been reported in greater detail elsewhere (5).

## Tar Sands Derived Fuel

Bitumen obtained from the Athabasca (Canadian) deposits was mined and refined by Great Canadian Oil Sands, Ltd. (GCOS). Mined tar sands are treated with basic hot water and the bitumen was isolated by froth flotation. The crude bitumen was coked (900-1000°F) and the distillates fractionated. The straight run middle distillate from coking was hydrotreated ( $\sim$ 1500 psig H<sub>2</sub>; 700-750°F). The hydro-treated middle distillate, known as Unifined Kerosene (herein designated "Tar Sands") is usually used for blending stock. The major process steps employed are shown schematically in Figure 3. Details of the processing of Canadian tar sands have been reported elsewhere (6).

## Petroleum Derived Fuels

The petroleum derived fuels used in this work were typical JP-5 grade fuels which passed all Navy specifications for JP-5 grade fuels and were typical of the fuels used at the Naval Air Propulsion Test Center. The history and details of refining of these fuels were not available.

## Fuel Properties

The physical properties of all fuels used in this study were determined according to Navy specification MIL-T-5624J, using ASTM standard methods. Heats of combustion, freeze points, flash points, smoke points, viscosities, as well as *n*-alkane composition for these fuels are reported in reference (1). Complete tabulations of physical properties for all fuels used in this study have been reported (3, 4, 7, 8).

## Results and Discussion

### FMR Analysis

The aromatic fraction of each fuel was isolated by column chromatography over activated silica gel. The aromatic fractions, which were cleanly separated from the fuel saturates, were combined and the solvent removed under reduced pressure on a rotary evaporator. The aromatic concentrate was diluted approximately 1:3 (vol:vol) with spectrograde CCl<sub>4</sub> and the 60 MHz FMR spectrum recorded. The FMR spectrum of a typical JP-5 grade fuel aromatic fraction is shown in Figure 4. The areas under the peaks which correspond to structurally distinct protons were determined and the data analyzed according to the method of Clutter and co-workers (9). The results of this analysis describe the fuel aromatic fraction in terms of the "average molecule." The calculated parameters which are presented in Table I are defined below:

### Average Molecular Parameter

<u>Symbol</u>	<u>Definition</u>
# CA	Average number aromatic carbons per average molecule
# naphthene	Average number naphthene (tetralin) rings per average molecule
MW	Average molecular weight
# alkyl gps	Average number alkyl substituents per average molecule
n	Average number carbons per alkyl substituent
% mono	Average percent monocyclic aromatics in sample

TABLE I

AVERAGE MOLECULAR PARAMETERS OF JP-5 GRADE  
FUEL AROMATICS FROM PMR DATA

Fuel	# CA	# NAPHTHENE	Parameters <sup>Ⓐ</sup>			% MONO	FORMULA
			MW	# ALKYL GPS	n		
Sun 1	6.65±0.05	0.75±0.05	179.7±2.8	3.05±0.05	2.25±0.15	84.3±2.0	C <sub>13.5</sub> H <sub>17.4</sub>
Sun 2	6.4±0.0	0.65±0.05	177.9±3.6	3.15±0.05	2.20±0.1	89.8±0.9	C <sub>13.4</sub> H <sub>17.8</sub>
Aged Sun 1	6.5±0.1	0.7±0.0	175.9±2.4	3.1±0.0	2.15±0.05	86.8±2.4	C <sub>13.3</sub> H <sub>17.1</sub>
Aged Sun 2	6.6±0.2	0.6±0.0	174.0±0.5	3.25±0.05	2.05±0.05	86.7±3.3	C <sub>13.1</sub> H <sub>16.9</sub>
Sun 5	6.5±0.2	0.45±0.05	156.5±1.3	2.7±0.1	1.95±0.05	87.7±4.2	C <sub>11.8</sub> H <sub>14.9</sub>
Sol. Extr.	6.55±0.05	0.70±0.0	191.5±6.3	3.25±0.05	2.40±0.1	87.0±1.5	C <sub>14.3</sub> H <sub>19.4</sub>
Paraho <sup>Ⓛ</sup>	6.8±0.3	0.35±0.5	172.5±4.1	2.95±0.05	2.05±0.05	80.0±6.5	C <sub>13.0</sub> H <sub>16.8</sub>
Tar Sands	6.45±0.05	0.35±0.05	165.9±2.9	3.0±0.2	2.0±0.0	87.6±1.3	C <sub>12.5</sub> H <sub>16.3</sub>
Petroleum-1	7.1±0.2	0.4±0.0	172.1±3.9	2.85±0.05	2.1±0.2	74.6±3.7	C <sub>13.0</sub> H <sub>16.6</sub>
Petroleum-2 <sup>Ⓢ</sup>	7.3	0.3	171.1	2.9	1.9	68.4	C <sub>12.9</sub> H <sub>15.9</sub>

<sup>Ⓐ</sup> Calculated from 60 MHz PMR spectrum according to reference (9). Average parameters ± average deviation of two runs shown, unless specified otherwise.

<sup>Ⓛ</sup> Average of three runs.

<sup>Ⓢ</sup> Average of one run.

In a separate set of experiments, some coal derived fuels were subjected to mild but prolonged heating (110°F; 6 months) to ascertain storage stability. The aromatic fractions of the Sun 1 and Sun 2 fuels, so treated, were isolated and subjected to FMR analysis in an effort to observe changes in composition upon storage. The results of analysis of the aromatic fractions of these fuels ("aged Sun 1," "aged Sun 2") are also reported in Table I.

Before a detailed analysis of the results in Table I is made, a discussion of sources of error in the data is necessary. There are a number of potential sources of error: incomplete resolution of the aromatic fractions from other fuel components; loss of low boilers during concentration of the column chromatography fractions; incorrect estimation of the average parameters caused by using a narrow cut aromatic fraction. Column chromatography over activated silica gel is a well known technique which has been widely used for separation of fuel components (10). However, it has recently been called to our attention that *n*-paraffins, particularly in higher boiling fractions, can tail into the aromatics fractions during chromatography over silica gel (11). The data in Table I indicates that such processes are not detectable by FMR if they are occurring at all. For instance, the Paraho fuel, which is almost 40% *n*-paraffins (1), shows a low value for *n* (average number of carbons per substituent). If appreciable quantities of paraffins were tailing into the aromatic fractions during the chromatographic separation, this parameter would be much higher. It would not be required to have a large concentration of *n*-hexadecane, for instance, in the separated aromatics in order to observe a large increase in *n*. Furthermore, the values for *n* found for all fuels vary only slightly from run to run (approximately 5%). We would expect a larger variation for the *n* parameter if incomplete separation were occurring.

Loss of low boiling aromatic components during concentration of the individual fractions was also of concern to us. However, the data in Table I indicate that this process is unimportant. Loss of low boilers would result in a higher "apparent" concentration of naphthalenes. The % mono data in Table I shows little variation between runs for most fuels.

Lastly, the method of Clutter and co-workers (9) may incorrectly estimate some average parameters if narrow cut fuels are used (12). Qualitative analysis by glpc of some of the aromatic fractions on a 10' X 1/8" OV-225 column indicates the presence of at least 60 distinct peaks which are not resolved (implying presence of related structural isomers). We therefore feel the calculated parameters in Table I correctly reflect the composition of the aromatic fractions. A complete discussion of the advantages of the Clutter method, compared to others available, has been given elsewhere (9).

In another control experiment, the molecular weights calculated from FMR data (Table I) were compared to those determined by vapor phase osmometry (VPO) (13) and by mass spectroscopy (MS) (14). A comparison of the molecular weights determined by the three methods is presented in Table II below.

TABLE II  
COMPARISONS OF MOLECULAR WEIGHTS OF AROMATIC FRACTIONS

<u>Sample</u>	<u>Molecular Weight</u>		
	<u>VPO</u>	<u>MS</u>	<u>FMR</u>
Sun 1	194	179	176
Sol. Extr.	240	206	198
Sun 5	175	-	158
Paraho	189	171	176
	195	-	168
Petrol. JP-5	195	-	176

The VPO molecular weights indicate that the PMR method correctly reveals changes in molecular weights between samples. The mass spectrometric molecular weights correspond very closely to those obtained by PMR. These data imply that the PMR method generates accurate molecular weights for these aromatic fractions. By analogy, the other parameters listed in Table I are at least as accurate as the molecular weight.

The data in Table I suggest a fairly clear picture of the nature of the aromatic fractions of these fuels. The average molecular weight of Sun 1 and Sun 2 (new and aged), Paraho, Tar Sands, and both petroleum fuels, Pet-1 and Pet-2, is 174.5 (Table I). Only two fuels, Sol. Extr. and Sun 5, had molecular weights which differed markedly from this average. The former fuel had the highest distillation end point ( $>287^{\circ}\text{C}$ ) of all fuels, while the latter fuel had the lowest distillation end point ( $261^{\circ}\text{C}$ ). Most fuels had between 6.5 and 7 aromatic carbons, approximately 3 alkyl groups and 2 carbons per alkyl group in their average aromatic molecule. This similarity in the nature of the aromatic fractions is noteworthy since the fuels examined were derived from starting materials which are chemically, as well as geologically, different.

While the aromatic fractions of these fuels are similar in overall features, there are important differences. For instance, the widest variation in the value of any parameter was observed for # naphthene. It must be recognized that for tetralin (1,2,3,4 - tetrahydronaphthalene), there is one (1.0) naphthene ring per molecule. In Table I, the # naphthene, of the aromatic fractions, varies from 0.75 and 0.7 (Sun 1 and Sol. Extr.) to 0.3 (Pet-2). This fundamental difference in the aromatic fractions might account for differences in the viscosities of the whole fuels (17,18). There are also variations in n, # CA, and # alkyl gp between these fuels. For instance, one might have apriori assumed that the Sun 1 and Sun 2 fuels would exhibit a high # CA due to the polycondensed aromatic nature of coals (15). However, the # CA is higher for both petroleum derived fuels used than for any of the other non-petroleum derived fuels. This is also reflected in the % mono values. The % mono is lowest for the petroleum derived fuels, which implies more naphthalenes in the petroleum fuels than in the other fuels.

This apparent anomaly, that the coal derived fuels contain less dicyclic aromatics, can be made understandable if we consider the naphthene content of these aromatic fractions. The naphthenes present in these aromatic fractions are of the tetralin-indane type. Clearly, these naphthenes are derived from the parent dicyclic aromatic by hydrogenation. The aromatic protons of these naphthenes contribute to the area of monocyclic aromatics (7.05 - 6.6 ppm; Figure 4), while the # naphthene is calculated from the integrated area under the protons  $\beta$  to the aromatic ring of tetralin or indane (1.9 - 1.65 ppm; Figure 4). Since vigorous hydrotreatment will convert naphthalenes to tetralins, we should ideally compare % mono (or # CA) between fuels which have undergone equivalent processing. This process data was not available for the petroleum derived fuels. Instead, we can compare the total amount of dicyclics (naphthalenes + tetralins-indanes) between fuels.

The percent of dicyclic compounds in these fractions can be calculated to a first approximation in the following manner:

$$\% \text{ Dicyclics} = (\% \text{ mono}) (\# \text{ naphthene}) + (100 - \% \text{ mono}) \quad 1)$$

If the results in Table I gave a value of 1.0 for # naphthene, then each aromatic molecule would contain a tetralin or indane type ring. Hence, values less than 1.0 for # naphthene indicate the fraction of the total number of aromatic molecules which are of the tetralin-indane type. For instance, in the case of the Sun 1 fuel, 75% of the molecules are tetralins or indanes (i.e., # naphthene = 0.75). Since the aromatic protons of these naphthenes are counted as "benzenes," we must correct the % mono figure as shown in equation 1). The last term on the right hand

side of equation 1) is the % naphthalenes in the sample. By working through this equation, we find that 78.9% of the aromatic molecules in the Sun 1 fuel are dicyclic ( $84.3 \times (0.75) + (100-84.3)$ ). Similarly, 68.5% of the aromatic molecules are dicyclic in the Sun 2 fuel. However, the % dicyclic aromatic molecules for the Pet-1 and Pet-2 fuels was found to be 55.2 and 52.1, respectively. Hence, the coal derived fuels have significantly more dicyclic (condensed) ring structures than the petroleum derived fuels. As expected, the coal derived fuels contained less naphthalenes than the petroleum derived fuels, because of the vigorous hydrotreatment to which they were subjected.

In addition to correctly predicting the order of molecular weights (Table II), the FMR method detects changes in other parameters which are consistent with changes in fuel properties. For instance, Sun 1 and Sun 5 were fuels produced from a Western Kentucky coal via an identical sequence of process steps but the distillation end point of Sun 1 was 279.5°C, while the distillation end point of the Sun 5 was 261°C. The lower distillation end point of the Sun 5 fuel is clearly reflected in the properties of the Sun 5 aromatics: lower values for n, MW, # alkyl gp compared with those values for Sun 1.

While large changes in fuel aromatics are clearly detectable by the FMR method employed, we could not detect changes in the aromatic fractions after a storage stability test (Table I). Under the static, oxygen deficient conditions of the storage test, aromatic hydroperoxide which forms would not decompose rapidly. The aromatic hydroperoxides would be separated from unoxidized compounds upon silica gel chromatography (16). The most oxygen labile aromatics would therefore be removed from the remainder of the aromatic "pool." There are small and irregular changes in the average parameters of aged Sun 1 and Sun 2. These changes are probably insignificant, although a statistical analysis was not performed. The latter conclusion is consistent with the observation that no gums or sediment were observed in the aged Sun 1 and aged Sun 2 fuels and furthermore, that the thermal stability of both aged fuels was unchanged.

#### Correlation of Average Parameters with Fuel Properties

The correlation of fuel properties with compositional data has been an active area of research for some time (19-21). Our interest in this area was not to develop a set of equations from which one can calculate a specific fuel physical property from compositional data (21), but rather to attempt to explain some of the sub-specification properties of these alternate fuels. Some properties which were of concern to us were the smoke point, viscosity and freeze point (1).

In general, fuel properties were plotted against n, # CA, # alkyl gps, # naphthene. Each individual parameter, combinations of parameters (i.e., # CA/n X # alkyl gp) and parameters which can be calculated from the data in Table I, such as wt. % H (aromatics) and approximate diameter of the average aromatic molecule, were plotted against the fuel property of interest. When straight line relationships were observed, least squares regression analysis was used to calculate the coefficient of correlation, r. It should be emphasized that baseline data for a representative sampling of petroleum JP-5 grade fuels has not yet been obtained. Selected fuel property-FMR parameter correlations, which are typical of our results, are illustrated below.

It has been shown that fuel smoke point is controlled by the aromatic content (vol. %) of the fuel (1). This correlation, as well as others (19, 22), suggest that some property of fuel aromatics controls combustion characteristics. However, we could find no correlation between fuel smoke point and any property of fuel aromatics. Two examples, which are typical, are shown in Figures 5 and 6.

Fuel viscosity is a fundamental property which directly controls atomization in combustors and affects fuel control equipment. It is known that viscosity of liquid mixtures can often be represented by a simple additivity law (25). In addition, the viscosity of cycloparaffins, as well as tetralins and indanes, can differ widely from the viscosity of the parent aromatic (17, 18). It was therefore somewhat surprising that the fuel viscosity was found to correlate so closely with the "diameter" of the average aromatic molecule (Figure 7). The diameter of the average aromatic molecule was estimated as follows:

$$\text{Diameter (A)} = \text{Fr. mono (2.8)} + \text{Fr. DI (4.9)} + n(0.89) \text{ (\# alkyl gp)} \quad (2)$$

where Fr. mono = Fraction monocyclic rings  
Fr. DI = Fraction dicyclic rings, including tetralins (from equation 1))

In these JP-5 grade fuels, we may be dealing with a special phenomenon. When lube oil fractions are hydrotreated, the viscosity of the oil decreases (17). However, the graph in Figure 7 implies that the viscosities of the aromatics and the tetralins and cycloparaffins derived therefrom are similar. This view is supported by the negligible change in fuel viscosity upon vigorous hydrotreatment of the Sun 1 and Sun 2 fuels (3), which resulted in nearly complete dearomatization.

Dimitroff and co-workers (16, 17) have shown fuel freezing to be a complex process with *n*-alkanes playing an important role, but with all fuel components interacting during freezing. Hazlett and co-workers (1) determined that fuel freeze point is related to the concentration of *n*-hexadecane but not the total *n*-alkane concentration for these alternate fuels. It can be shown (26) that fuel freeze point probably does not follow a simple additivity rule. In Figure 8, a plot of fuel freeze point against the diameter of the average aromatic molecule is shown. The good correlation degrades markedly when the volume % aromatics (in each fuel) is accounted for (Figure 9). Hence, fuel freeze point seems to depend more strongly on the size rather than the quantity of aromatics present in fuel. One possible explanation for this behavior is that the larger aromatics cause an ordering of non-aromatic fuel components in a way similar to the ordering of water by dissolved soaps. As shown in Figure 8, the Paraho fuel (2.45% *n*-hexadecane) does not correlate well but the urea extracted (1) Paraho fuel (0.17% *n*-hexadecane) does fall near the line. Work is continuing on a variety of petroleum derived JP-5 grade kerosene in order to establish baseline data.

#### Acknowledgement

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Fig. 1  
PROCESSING SCHEMATIC FOR SUN COAL DERIVED FUELS

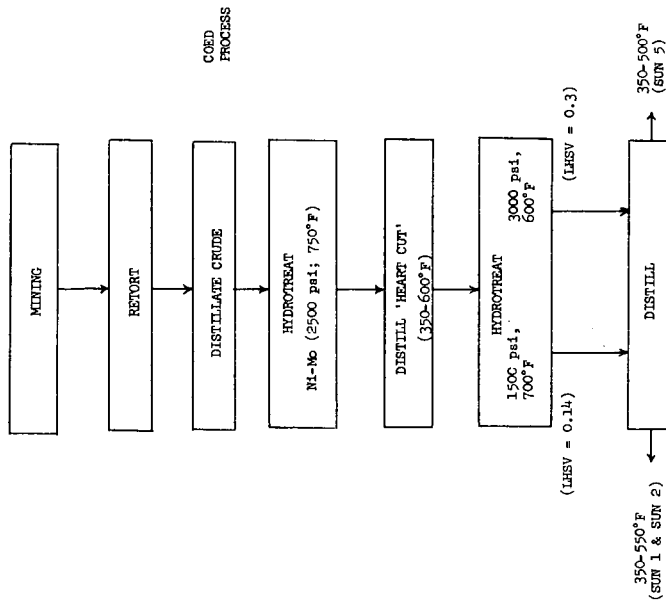


Fig. 2  
PROCESSING SCHEMATIC FOR PARAHIO FUEL

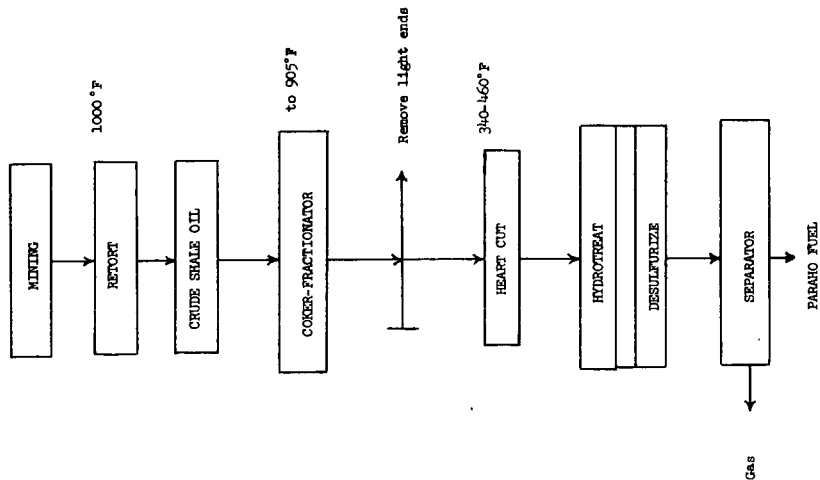




Fig. 3  
PROCESSING SCHEMATIC FOR TAR SANDS FUEL  
("UNIFIED KEROSENE")

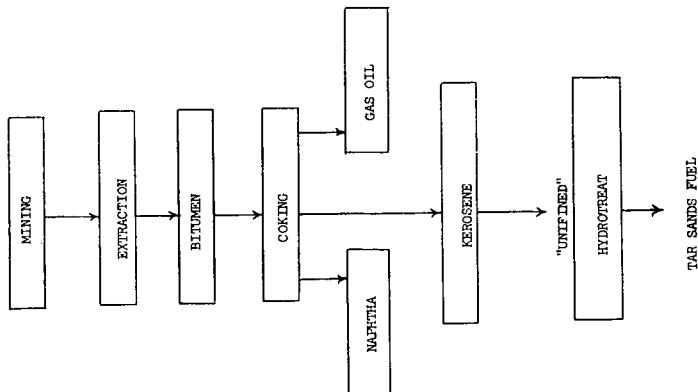


Fig. 4  
60 MHz PMR Spectrum of Kerosene Aromatic Fraction

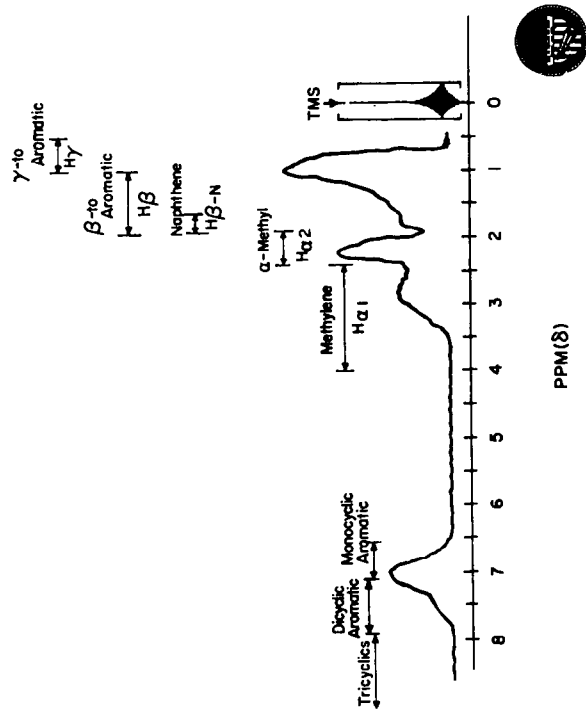


Fig. 1

PROCESSING SCHEMATIC FOR SUN COAL DERIVED FUELS

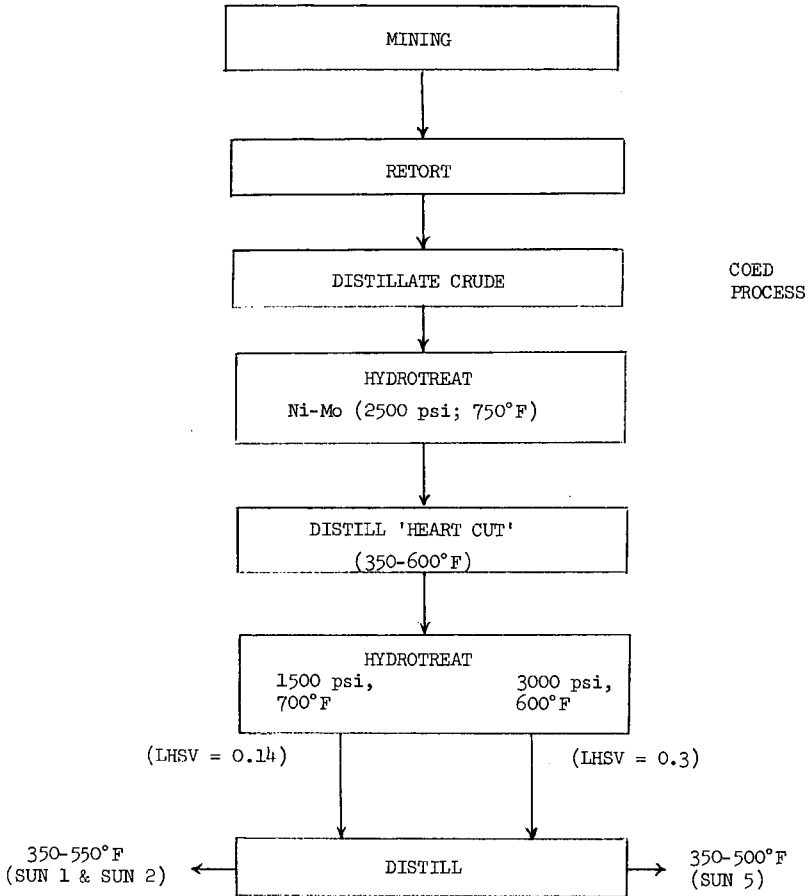


Fig. 2  
PROCESSING SCHEMATIC FOR PARAHO FUEL

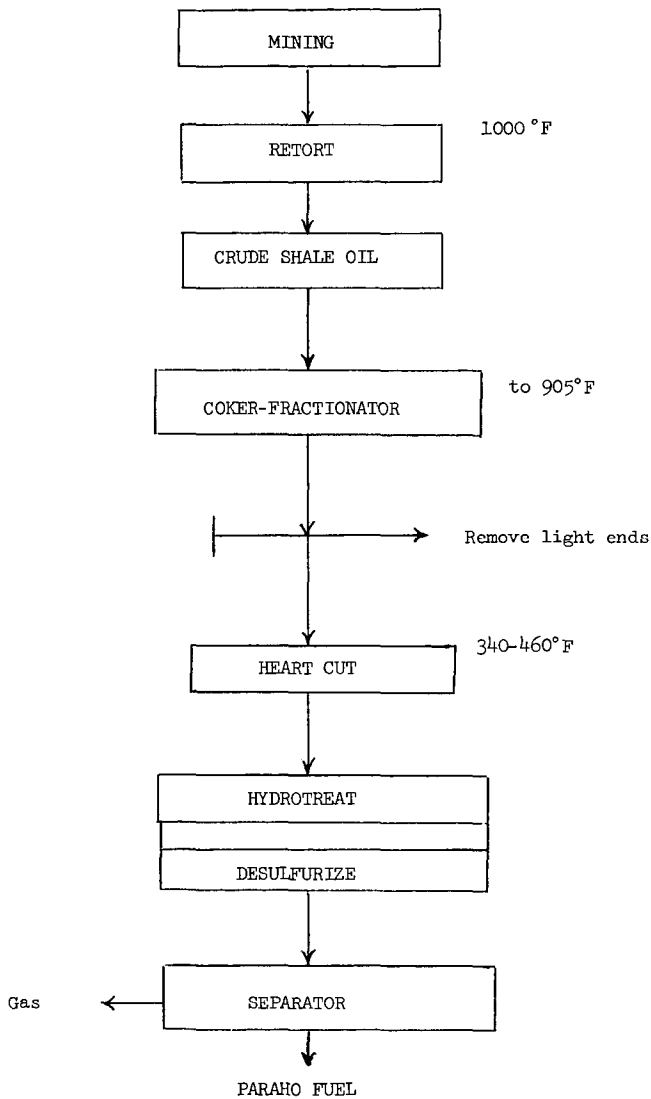


Fig. 3

PROCESSING SCHEMATIC FOR TAR SANDS FUEL  
("UNFINED KEROSENE")

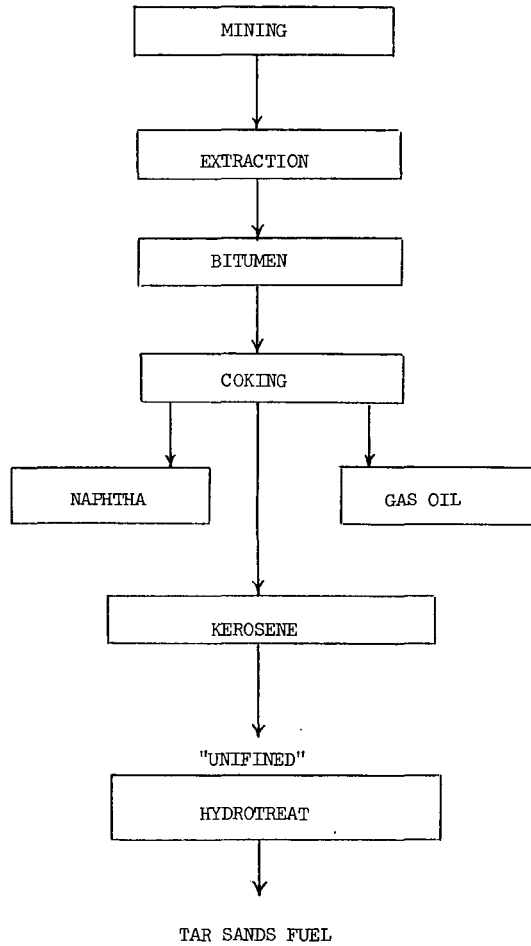


Fig. 4

# 60 MHz PMR Spectrum of Kerosene Aromatic Fraction

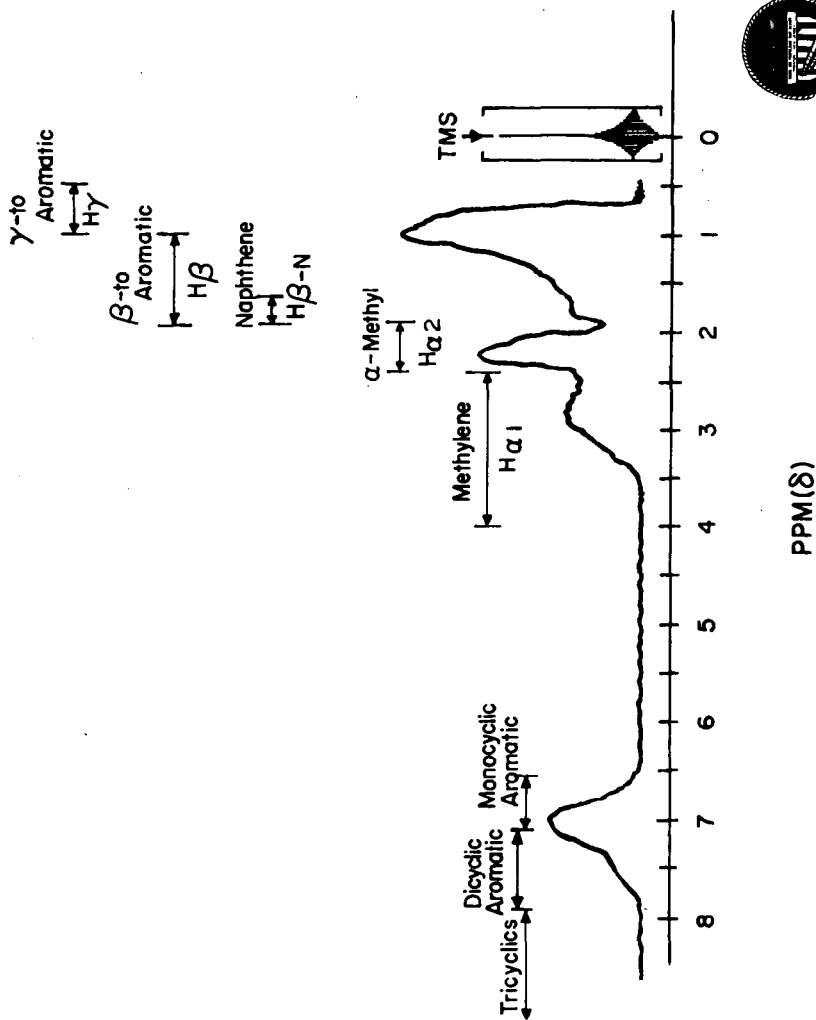


Fig. 5  
FUEL SMOKE POINT VS. AROMATICITY

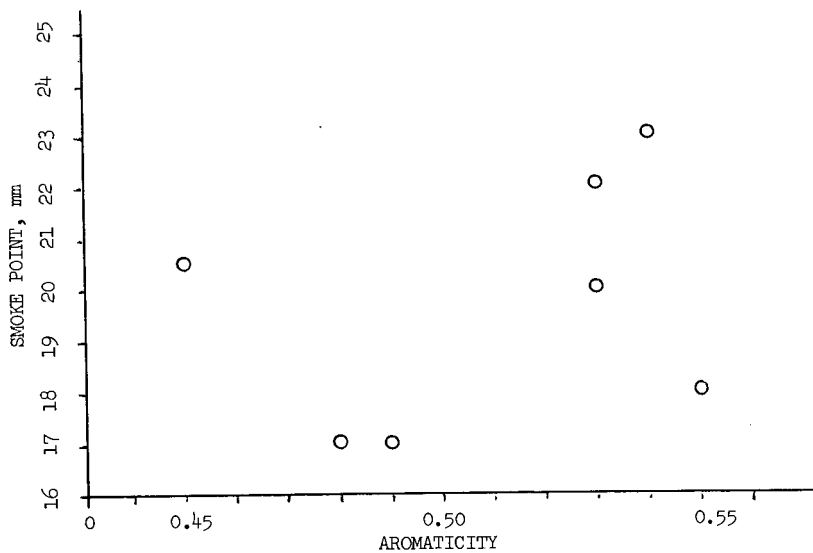


Fig. 6  
FUEL SMOKE POINT VS.  $\text{Wt \% H}_{(\text{AROM.})} \times \text{Vol \% AROMATICS}$

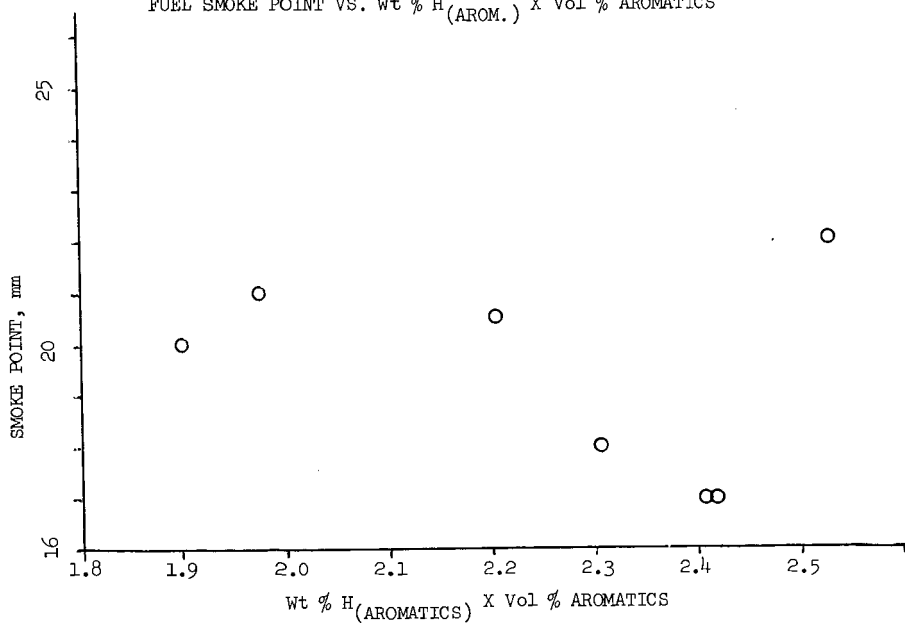


Fig. 7

FUEL KINEMATIC VISCOSITY ( $-30^{\circ}\text{F}$ ) VS. DIAMETER OF AVERAGE AROMATIC MOLECULE

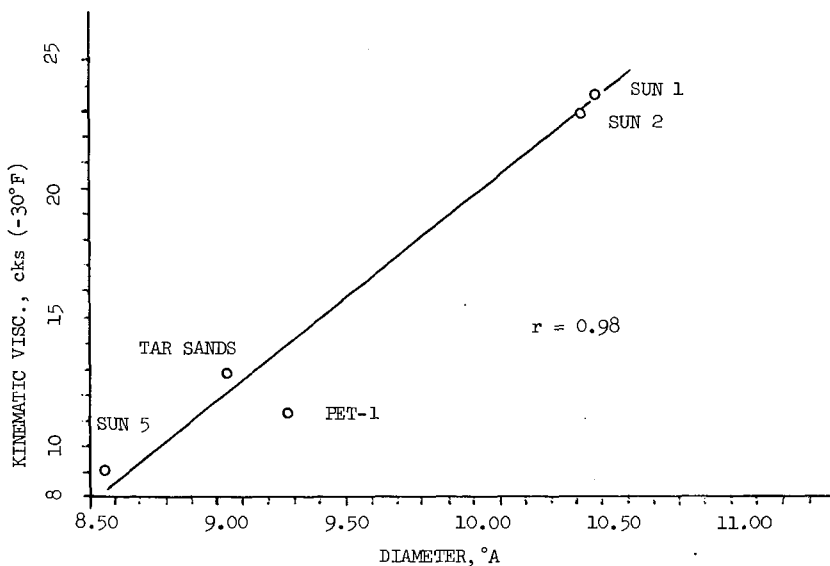


Fig. 8

FUEL FREEZE POINT VS. DIAMETER OF AVERAGE AROMATIC MOLECULE

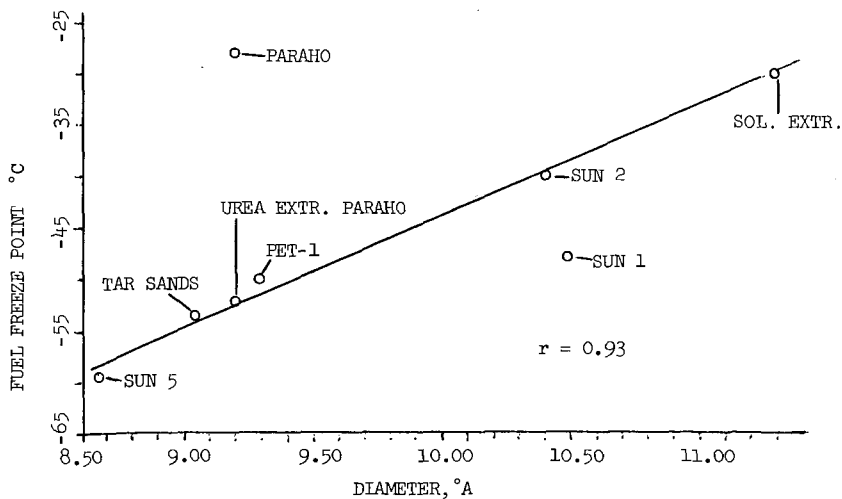
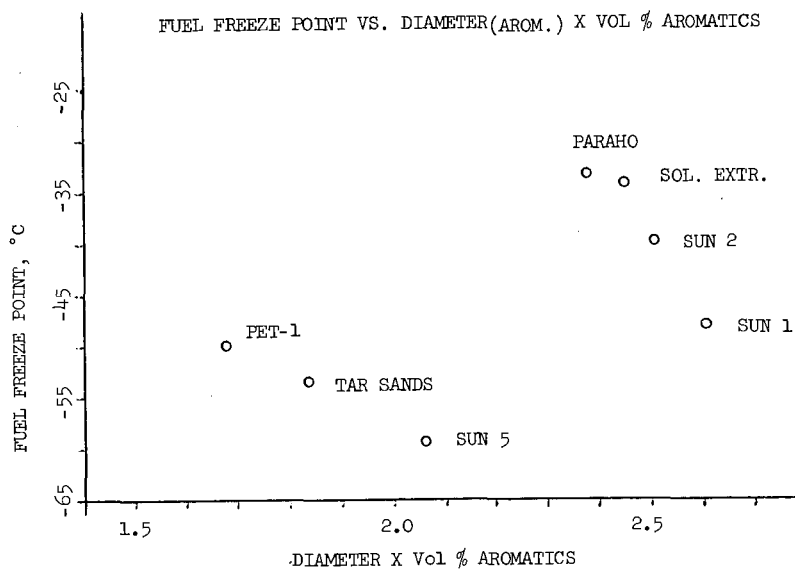


Fig. 9





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